Report No. TR 80-F-4

MANUFACTURING METHODS AND TECHNOLOGY
(MANTECH) PROGRAM

MM&T MANUFACTURING METHODS FOR GRADIENT FURNACE PROCESSING OF CERAMIC ARMOR AND STRUCTURAL CERAMICS

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FINAL REPORT

U.S. ARMY AVIATION RESEARCH AND DEVELOPMENT COMMAND

This project was accomplished as part of the US Army Aviation Research and Development Command Manufacturing Technology program. The primary objective of this program is to develop, on a timely basis, manufacturing processes, techniques, and equipment for use in production of Army materiel. Comments are solicited on the potential utilization of the information contained herein as applied to present and/or future production programs. Such comments should be sent to: US Army Aviation Research and Development Command, ATTN: DRDAV-EGX, 4300 Goodfellow Blvd., St. Louis, MO 63120

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ABSTRACT

The growth of large single crystal materials opens up many possibilities for new applications of ceramics, such as in the case of transparent armor, lasers, and laser windows.

Single crystals of sapphire have been made by many techniques, the best known probably being the Verneuil method for the production of jewels and bearings. Until recently, the largest crystals of sapphire were probably those obtained by the Czochralski technique where crystals up to three inches in diameter were grown.

At the Army Materials and Mechanics Research Center (AMMRC) a coordinated in-house effort has been underway to develop an improved approach to the growth of large uniform single crystals of sapphire. The main effort centered about a research and development project on the gradient furnace synthesis of advanced ceramics.

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INTRODUCTION

The growth of large single-crystal materials opens up many possibilities for new applications of ceramics such as in the case of transparent armor, lasers, and laser windows. With the increase in size of single crystals, many opportunities for cost reduction arise, as has been demonstrated in the semiconductor industry with the production and processing of large quantities of uniform wafers.

Single crystals of sapphire have been made by many techniques, the best known probably being the Verneuil method for the production of jewels and bearings. Until recently, the largest crystals of sapphire were probably those obtained by the Czochralski technique where crystals up to three inches in diameter were grown. Variations of this technique were developed by Tyco[1] to produce long and uniform crystals which would be more amenable to cost-effective engineering applications.

At the Army Materials and Mechanics Research Center (AMMRC) a coordinated effort has been underway in 6.1, 6.2, and MM&T programs to develop an improved approach to the growth of large, uniform, single crystals of sapphire. The main effort centered about a research and development project on the gradient furnace synthesis of advanced ceramics under D. Viechnicki and F. Schmid with supporting research characterization programs to provide X-ray, optical, and mechanical techniques to study the crystals and provide technical feedback. Schmid and Viechnicki consequently were able to demonstrate the effectiveness of the resulting crystal growth technique, called the gradient furnace technique[2,3] by growing single crystals up to six inches in diameter. It was this technique that consequently became the basis for this project.

The gradient furnace technique essentially consists of solidification of a melt onto a preexisting seed crystal contained in a high temperature crucible and cooled through controlled temperature gradients to obtain nucleation and crystallization of the melt into a single crystalline form. A schematic of the furnace used in this process is shown in Figure 1. In this particular process, helium gas was used to cool the crucible, although other gases or liquids may be substituted for helium.

The advantages of this process are:

a. The size restriction of the crystal is dependent only on the size of the crucible used in the furnace.

b. Various shapes can be made, dependent only on the crucible shape. The schematic showing the arrangement of the crucible and the material in the furnace is presented in Figure 2. The seed crystal is placed in the crucible above a heat exchanger which prevents the seed from melting during solidification by directing a flow of helium gas, or other suitable coolant, against the crucible beneath the seed. The crucible was heated from the sides by a graphite resistance heater and the flow of helium gas through the heat exchanger extracted heat from the crucible bottom beneath the seed. This cooling effect resulted in a temperature gradient which was the driving force for the radial growth of the crystal from the seed crystal. The temperature gradient can be controlled by adjusting the amount of helium flow through the heat exchanger.

1. MORRISON, A.D., et al. *Spinel Ribbon.* Tyco Laboratories, Inc., Contract DAAB05-72-C-5841, Final Report, July 1974.
2. SCHMID, F., and VIECHNICKI, D. *Growth of Sapphire Disks from the Melt by a Gradient Furnace Technique.* J. Am. Ceram. Soc., v. 53, no. 9, 1970.
3. SCHMID, F., and VIECHNICKI, D. *Apparatus and Method for Unidirectionally Solidifying High-Temperature Ceramic Material.* U.S. Patent 3,653,432, April 1972.

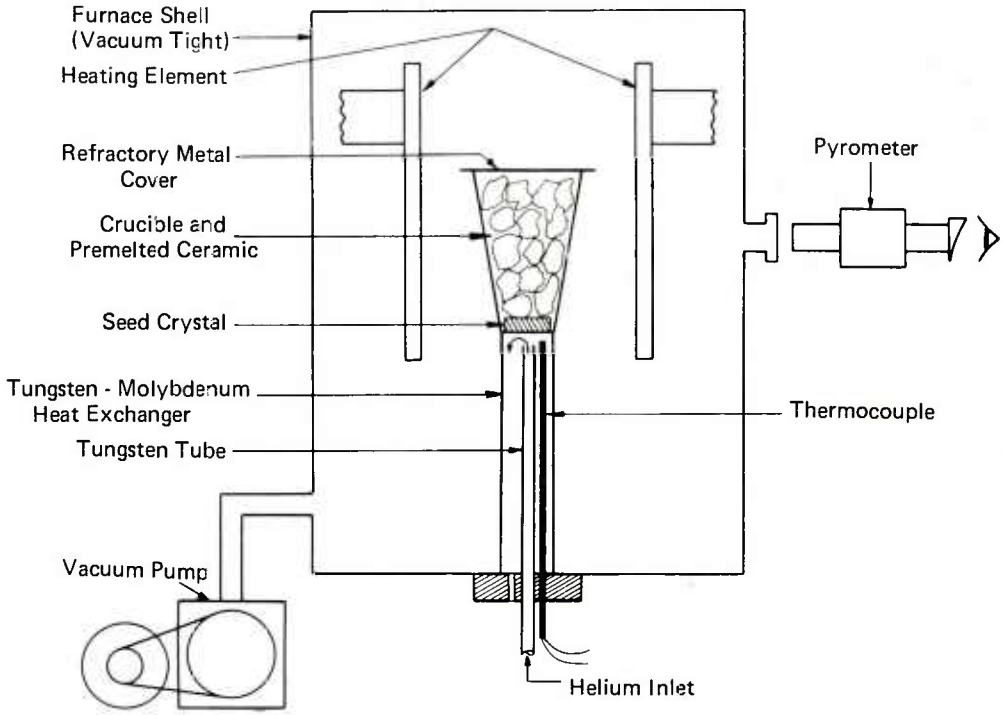


Figure 1. Schematic of gradient furnace.

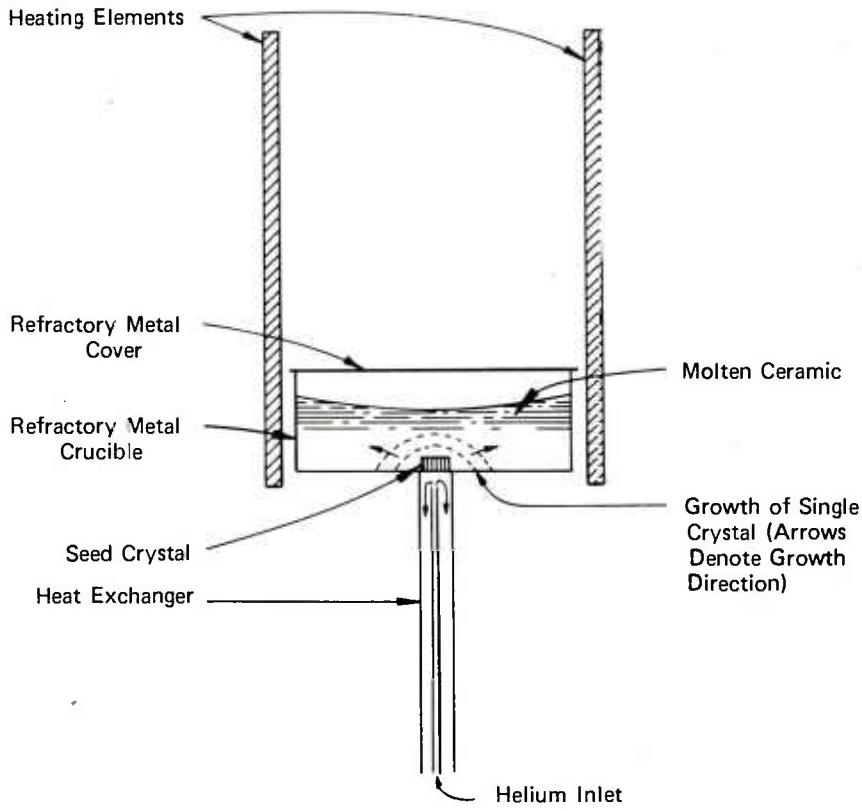


Figure 2. Schematic of arrangement in gradient furnace for growth of ceramic single-crystal disks.

OBJECTIVES

It has been demonstrated that sapphire single crystals up to 6-1/2 inches in diameter could be produced which have a rather high degree of optical transparency (Figure 3), and with other desirable properties such as a high modulus of elasticity (over 50,000 psi); resistance to extreme moisture, scratch, and rain erosion; light weight; and high hardness (9 on Mohs' hardness scale). Therefore it appeared that sapphire would be an excellent candidate as a transparent armor material. In order to provide an efficient source of sapphire for transparent armor, the goals of this project were, briefly:

1. the design and installation of a 20"-diameter furnace with an upper limit capability of an 18"-diameter sapphire single crystal;

2. growth of 12"-diameter sapphire single crystals;

3. establishment of experimental procedures so that single crystals could be reproducibly grown;

4. the establishment of cost-effective processing procedures for:

   a. cutting and polishing,

   b. recovery systems for helium,

   c. rapid testing of sapphire for optical quality, and

   d. developing reusable crucibles.

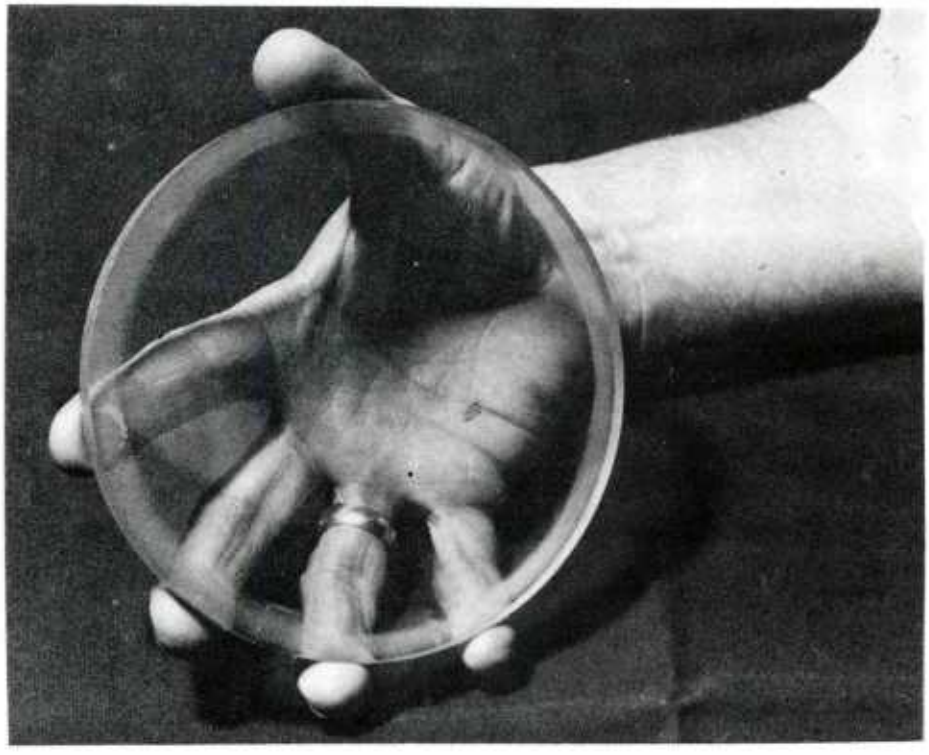


Figure 3. A 6-1/2"-diameter X 1/2"-thick polished sapphire single crystal disk.

CRYSTAL GROWTH

Background

The furnace used for the crystal growth procedure was a water-cooled vacuum furnace with a 20" ID graphite resistance heater as shown in Figure 4. The temperature controller consists of a saturable core reactor with a programmable console. The heating and cooling of the furnace was preprogrammed and controlled with the console shown in Figure 4b. The time-temperature program was predrawn on a mylar sheet and the sensing head followed the predrawn program, which in turn increased or decreased the power accordingly. The control console was used during the complete growth and cooling cycle of the sapphire crystal.

Crucibles used to melt the sapphire were made of molybdenum sheet spun into sizes which varied from 6-1/2" to 18" in diameter. A taper was designed into the crucible to change the gradient between the graphite resistance heater and the crucible.

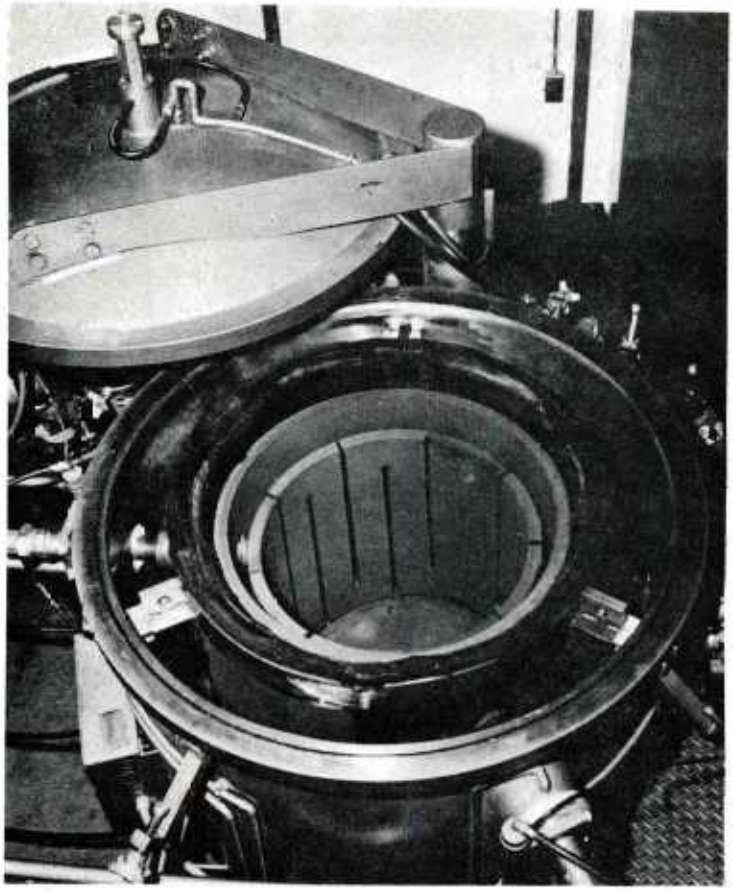
A helium recirculator was employed to remove heat from the crucible and thereby control the temperature of the seed crystal during growth. As shown in Figure 5, the helium recirculator system was charged from an external source to 15 psi. The two metal bellows pumps were arranged in parallel and pumped the gas into a tank through an MSA filter to an automatic flow control into an oxygen filter to remove any oxygen gas that may be in the system. The helium gas next passed through the heat exchanger, was cooled in a water cooling tower, and then filtered before being recirculated. With the use of the recirculator, the helium gas consumption was reduced from 30 or 40 cylinders per run to 1 cylinder per run.

Crackle (high-purity Al_2O_3) was placed in a molybdenum crucible containing a seed crystal which was located at the bottom center of the crucible. The crucible was placed above the heat exchanger in the water-cooled vacuum chamber as shown in Figure 1.

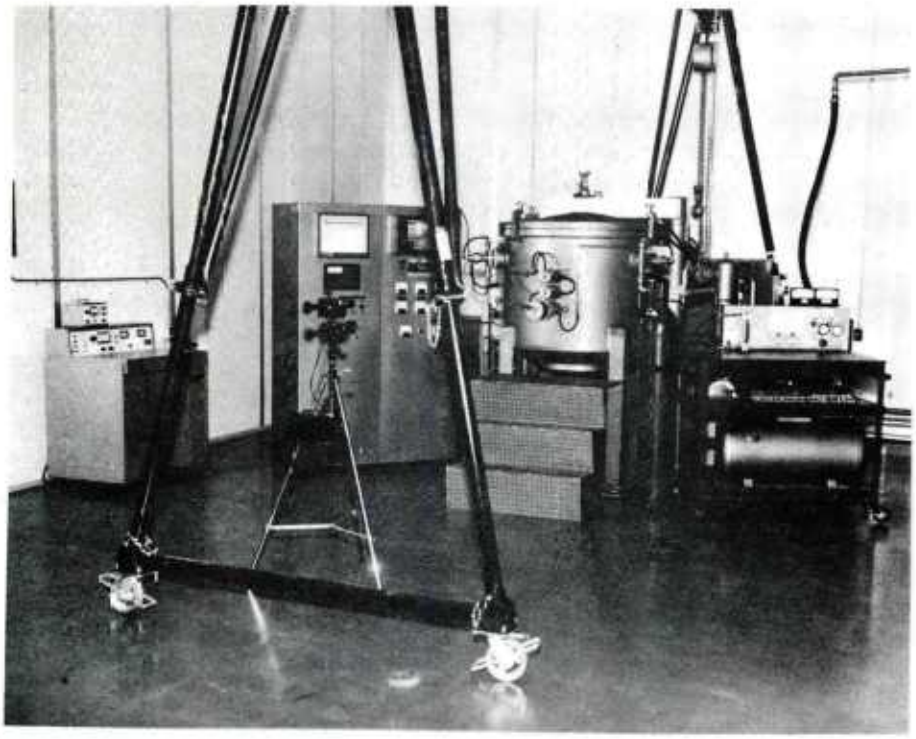
As the temperature of the furnace was increased to 2250°C, helium was recirculated into the heat exchanger to maintain the seed crystal at a maximum temperature of 1950°C. The temperature at the bottom of the crucible was continually monitored by a W5/W26% Re thermocouple. The temperature of 1950°C, approximately 100°C below the melting point of Al_2O_3, was maintained constant during the melting of the sapphire. After the sapphire as well as the surface of the seed crystal was completely melted, the helium gas flow was increased in order to raise the temperature gradient and start to solidify the new growth onto the seed crystal. The furnace was slowly cooled during the solidification process. The growth rate was approximately 1 centimeter per hour. Approximately 20 hours are required to grow an 8"-diameter crystal. The sapphire crystal was cooled at a rate of approximately 20°C per hour. In the supporting R&D study of the crystal growth of sapphire,* it was found that thermal oscillations due to fluctuations in electrical power or to changes in helium flow rate could cause thermal stresses which would "punch out" prismatic dislocation loops. Under special conditions, i.e., the approach of two rows of prismatic loops to one another, a crack could nucleate, resulting in the failure of the crystal growth run. Therefore, care was exercised to prevent such oscillations from occurring until the temperature of the ingot was below 1500°C. At the end of the cooling period, which lasted approximately 7 to 10 days, the furnace was opened and the crucible containing the sapphire crystal removed.

Many runs were made to insure that proper parameters were obtained so that the crystal growth could be duplicated.

*CASLAVSKY, J. L., and GAZZARA, C. P. *Characterization of Sapphire Single Crystals Grown by the Gradient Furnace Method*, unpublished.



(a) Cover removed to reveal graphite heater element.



(b) Helium recirculator and control console

Figure 4. Twenty-inch-diameter vacuum gradient furnace.

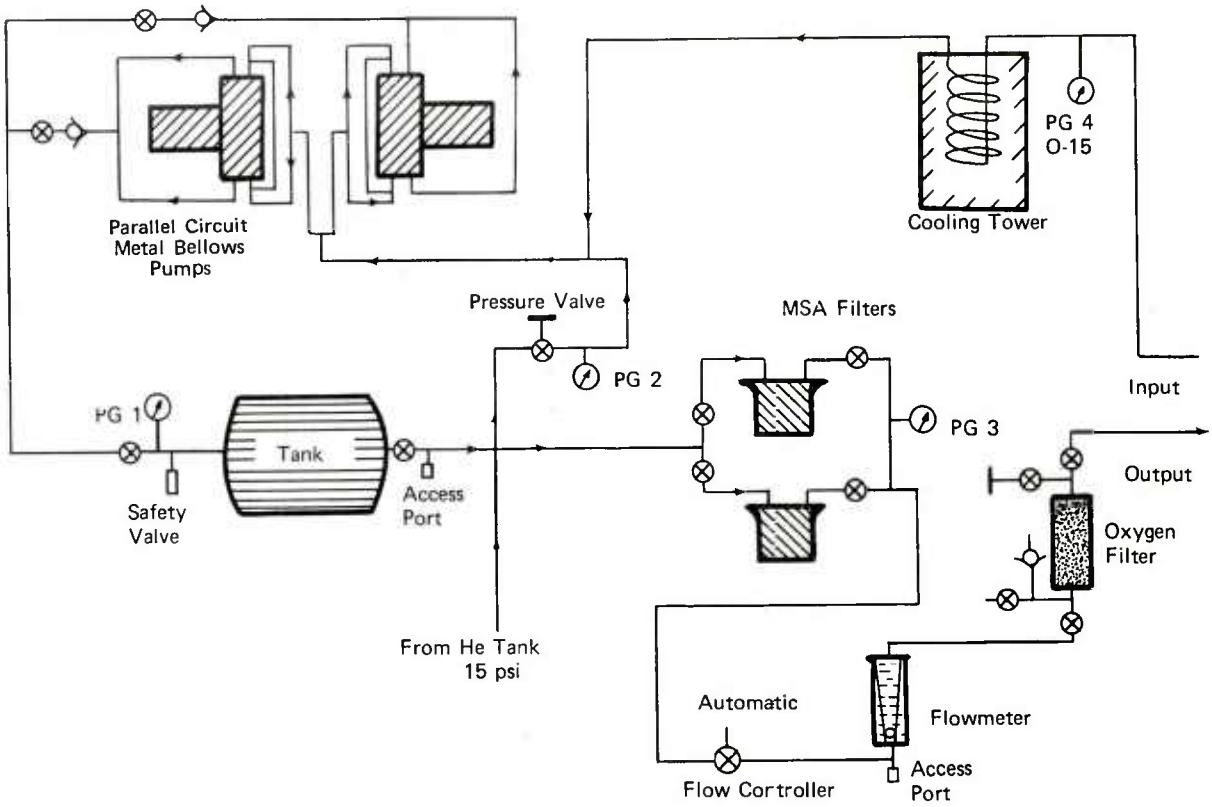


Figure 5. Schematic of a helium recirculation system used with gradient furnace.

POST GROWTH PROCESSING

Evaluation and Cost Effectiveness

Sapphire ingots were cut into disks with diamond cutting wheels, and diamond core drills were employed to cut cylindrically shaped columns out of the sapphire ingots. Diamond was used as a cutting medium due to the hardness of the sapphire. The cylindrical pieces were sliced to provide seeds for growing sapphire crystals and disks for evaluation. It was necessary to polish the disks prior to optical, infrared, or X-ray examination.

Polishing the sapphire disks to an optical finish required the use of a progression of finer grit sizes of diamond paste on a lapping apparatus. The present cost of such a time-consuming process is approximately $2.00 per square inch. A study of the effect of various diamond powder grit sizes on the transmission efficiency of infrared radiation was performed[4] and the results shown in Figure 6. It was concluded that the finer the grit size, the better the polish and the better the transmission up to an optimum grit size of 300. After a decrease in transmission beyond the 300 grit size, the transmission then increased again with increasing grit size. It is, therefore, of utmost importance that the surface finish of sapphire be specified, particularly when the radiation wavelength is long enough to result in large scattering losses and distortions.

4. DeLAI, A. J., GAZZARA, C. P., and KATZ, R. N. *The Production of Large Sapphire as a Potential Laser Window.* Proc. 4th Ann. Conf., IR Laser Window Materials, 19 November 1974, p. 475.

Because of the long time and high cost of polishing sapphire by conventional methods, an alternative approach to optical transparency seemed necessary. To produce an optically transparent armor plate, it was decided to coat the rough-cut sapphire surface with a smooth glass. The glass would adhere to the sapphire and produce an optically smooth surface. This technique was developed by A. D. Little, Inc.[5] A lead-base glass was formulated which had the same refractive index as sapphire. The sapphire plates, which were not transparent due to the existing rough surface, were dipped into the molten glass, resulting in a smooth transparent coating (Figure 7). In addition, the glass coating could be further polished by standard soft polishing techniques using Al_2O_3, B_4C, etc., affording a substantial cost saving without affecting the ballistic performance of the sapphire plate. Figure 8 shows the results of a cost analysis of the diamond finishing procedure as compared to the glass-glaze finishing process.

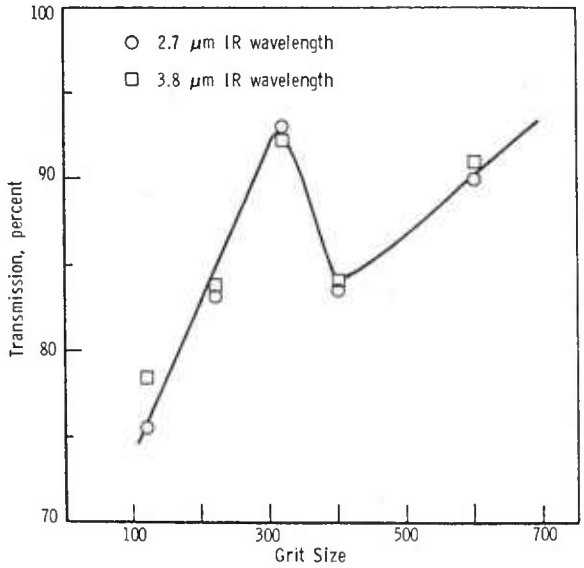


Figure 6. Infrared transmission curve of percent transmission versus wavelength through a polished 3/8"-thick sapphire single crystal disk.

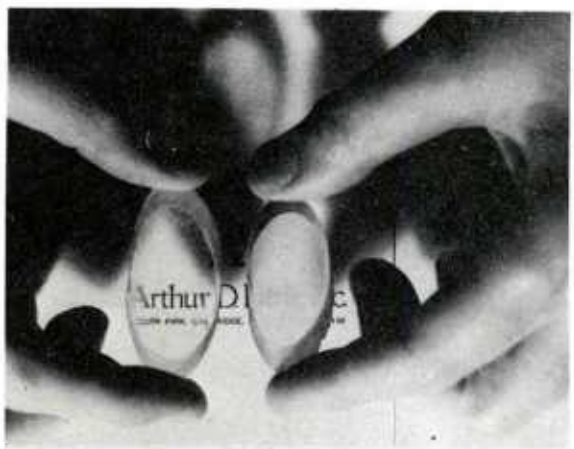


Figure 7. Photograph demonstrating transparency of as-cut glazed versus as-cut surface of sapphire.

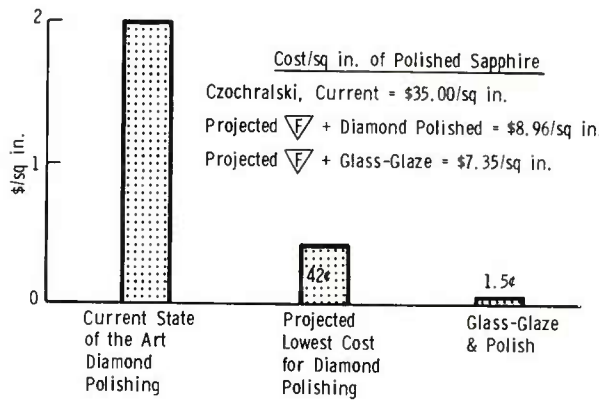


Figure 8. Cost comparison of finishing of glass-glaze sapphire to nonglazed sapphire.

5. HAGGERTY, J. S., and ROSSETTI, M. *Development of Inexpensive Finishing Process for Transparent Armor.* A. D. Little, Inc., Contract DAAG46-72-C-0170, Final Report, AMMRC CTR 73-34, September 1973.

Crystal Analyses and Characterization

1. Orientation

It was found by Caslavsky et al.[6] from a study of the slip systems in sapphire that for the configuration of growth crucible, the optimum seed orientation should be close to the $<01\bar{1}2>$ direction. Indeed, since using seed crystals close to such an orientation, the result has been a marked increase in the yield of crack-free single crystals of sapphire. In order to rapidly orient single crystal ingots of sapphire for cutting into seeds (1-1/4 to 2" dia.), a series of Laue back-reflection photographs have been compiled, together with stereographic projections, to develop a simple procedure for obtaining the optimum seed orientation.[7]

2. X-Ray Topography

An X-ray topographic investigation of basal dislocations in sapphire revealed the existence of Burger's vectors parallel to the $<2\bar{1}\bar{1}0>$ directions.[8] These results indicate the ambiguity associated with characterizing dislocation densities from etch pits, operative on dislocation outcrops, which are visible in optical micrographs. This observation has been verified[8] from the disparity in the areal densities of dislocations of planes with different orientations through a sapphire crystal. A three-dimensional display of the dislocations, unambiguously displayed in the X-ray topographs, affords a more accurate method of characterizing the true dislocation "density" in single crystals.

3. Dislocations

A more complete study of the dislocation behavior of sapphire revealed different forms which resulted from reactions and from passing through a characteristic progression of dislocation types. With increasing time-temperature conditions, dislocation tangles appear first, reacting to form helices and, with further heat treatment, transforming into loops.[9] A rather complete characterization of the final loop types, formed by heat treating sapphire, was performed* and provided not only a means of approximating dislocation densities but also yielded information as to the heat treatment history of the sapphire single crystal grown.

4. Pendelloesung X-Ray Fringes

The X-ray topographic procedure previously described required the slicing of thin transmission X-ray topographic specimens (approximately 100-mm thick) which were submitted to long-time X-ray exposures. Another X-ray technique was developed which made use of the observation of pendelloesung X-ray fringes. Basically, the greater the crystal perfection of the sapphire, the more intense and sharper the pendelloesung fringes would be. It was hoped that this technique could be developed as a nondestructive test to measure the degree of perfection of sapphire single-crystal slabs. Limited tests on sapphire single crystals obtained from different sources were performed to compare the perfection of the crystals. The sapphire crystals grown by this technique displayed pendelloesung fringes with the highest resolution thus far observed.

*CASLAVSKY, J. L., and GAZZARA, C. P. *The Investigation of the Strength of $\bar{g}.\bar{b}$. $X\,\bar{a}$ X-Ray Contrast from Prismatic Loops as a Function of ψ*, unpublished.

6. CASLAVSKY, J. L., SCHMID, F., VIECHNICKI, D., McCAULEY, J. W., and GAZZARA, C. P. *Method of Orienting Seed Crystals in a Melt and Products Obtained Thereby.* DARCOM Docket No. 7991, Serial No. 589317, filed in Patent Office, 23 June 1975.
7. GAZZARA, C. P., and CASLAVSKY, J. L. *An Intensity Modified Stereographic Projection of α-Al_2O_3.* Army Materials and Mechanics Research Center, AMMRC TR 71-26, August 1971.
8. CASLAVSKY, J. L., GAZZARA, C. P., and MIDDLETON, R. M. *The Study of Basal Dislocations in Sapphire.* Phil. Mag., v. 25, 1972, p. 35.
9. CASLAVSKY, J. L., and GAZZARA, C. P. *Dislocation Behaviour in Sapphire Crystals.* Phil. Mag., v. 26, 1972, p. 961.

5. Laue Spots

The third X-ray method employed to assess the perfection of sapphire crystals was simply to observe the width of Laue spots. Laue photographs were routinely taken on sapphire crystals to determine crystal orientation. Misorientation of grains over 1° could be observed. Some single crystals of poor quality would reveal Laue spots misoriented up to 3° in width.

6. Polariscope

An optical technique was set up to rapidly reveal the entire distribution and the presence of subgrains. This test exploits the optical anisotropy of sapphire.[10] By rotating the crystal under the crossed polarizers of a polariscope one is able to observe the individual subgrains as in Figure 9. With such a fast, economical, and effective test, crystals of sapphire, as grown, cut, or after polishing, were easily examined for the presence of subgrains.

7. Laser

Still another useful optical test, effectively and routinely employed, made use of a laser beam (Figure 10). When such a beam was passed through a sapphire crystal, scattering centers could be revealed. This test was especially effective for determining the amount of "veiling" in the finished sapphire disks (Figure 11). Such imperfections are caused by substitutional supercooling and could markedly affect the optical quality of sapphire for window applications.

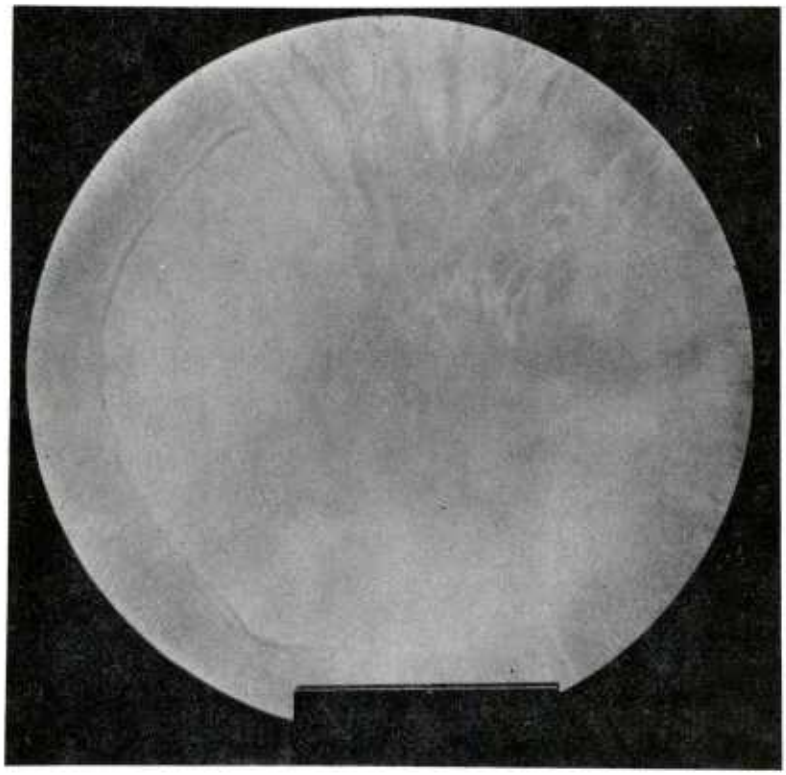


Figure 9. Polished sapphire single crystal disk photographed in polariscope, revealing subgrains.

10. McCauley, J. W. *Polariscopic Characterization of Sapphire and Spinel.* Army Materials and Mechanics Research Center, AMMRC TR 72-1, January 1972.

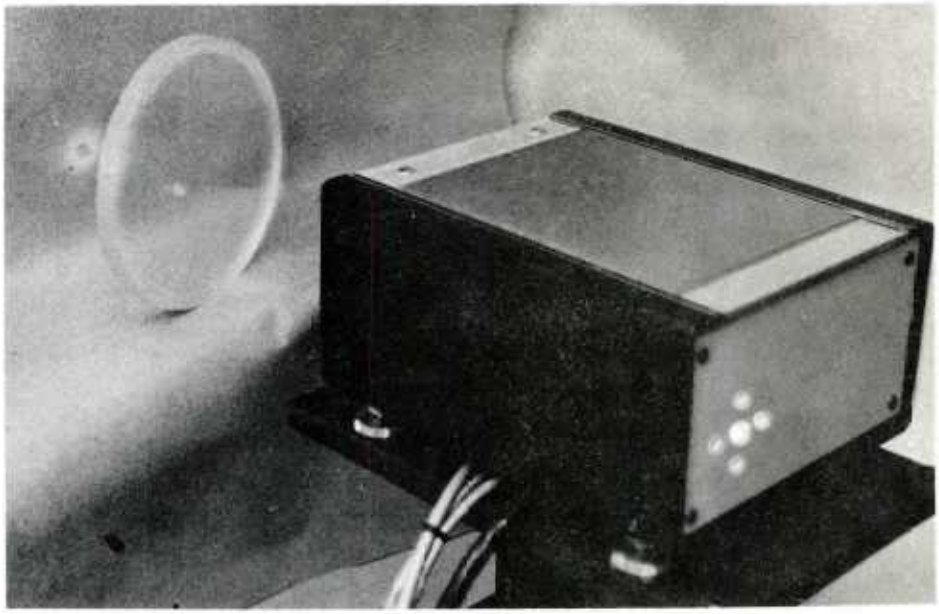


Figure 10. Laser beam passing through a sapphire single crystal disk.

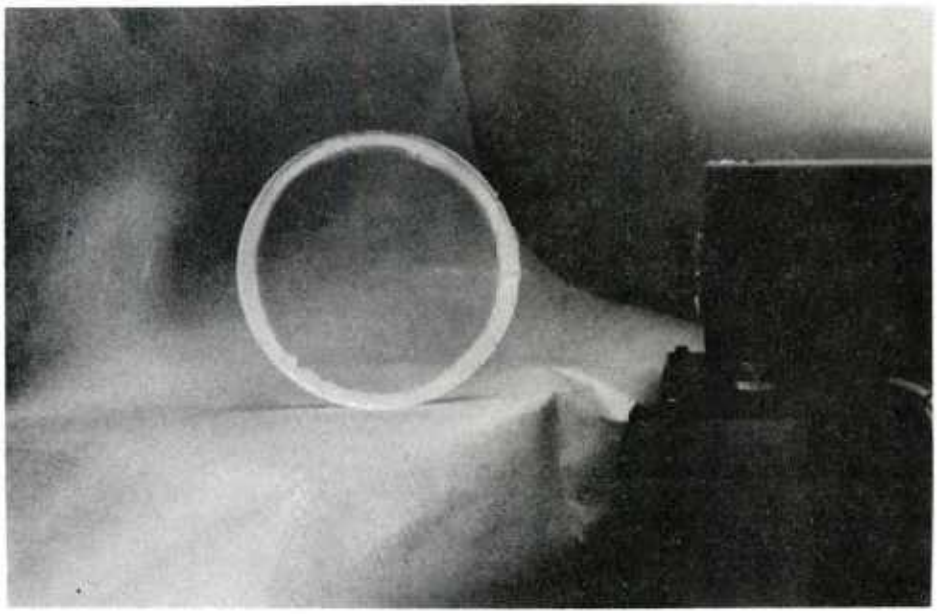


Figure 11. Laser beam passing through a sapphire single crystal disk (sideways) revealing "veiling."

8. Color

Another practical and sensitive type of optical test was simply observation of the color of the sapphire. A pink color was imparted to the sapphire when impure starting material (see Table 1) was used, or the crystal was contaminated with molybdenum. As the starting impurity levels were reduced, the sapphire crystals became clearer with a greater degree of optical transparency, and a water-white color was characteristic of those crystals held within close impurity tolerances.

9. Differential Infrared Spectroscopy

A characterization technique that was developed, mainly to test the transmission efficiency of infrared radiation or the absorptivity β, made use of a differential infrared spectrometer. The transmission curve, Figure 12, showed absorption peaks due to impurities in the sapphire and served as a test of the impurity levels. Neutron activation analyses were also performed to afford a check on the impurities in the sapphire single crystals and the results are listed in Table 2.

Table 1. COMPOSITION OF SAPPHIRE STARTING MATERIALS

Element	Concentration (ppm)	Element	Concentration (ppm)
B	0.02	Ti	0.3
N	3	V	0.04
O	Major	Mn	0.7
F	0.3	Fe	0.8
Na	3	Cr	0.4
Mg	0.3	Ni	0.2
Al	Major	Cu	2
Si	4	Zn	0.6
P	0.05	Y	≤0.5*
S	4	Mo	1
Cl	6	Rh	0.06
K	3	Ta	1†
Ca	0.7	Mg	12‡

Elements sought but not detected:

Li, Be, Ne, Sc, Co, Ga, Ge, As, Se, Br, Kr, Rb, Sr, Zn, Nb, Ru, Pd, Ag, Cd, In, Sn, Sb, I, Xc, Cs, Ba, La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Hf, Ta, W, Re, Os, Ir, Pt, Au, Ti, Pb, Bi, Th, U.

*Lines appear for this element but they may be due totally or partially to interference or may be absent. The value reported is the maximum that could cause the lines observed.
†This may be contamination from previous test samples.
‡On the surface only.

Table 2. NEUTRON ACTIVATION ANALYSIS OF A TYPICAL Al_2O_3 SINGLE CRYSTAL GROWN BY GRADIENT FURNACE TECHNIQUE

Element	Concentration (ppm)	Element	Concentration (ppm)
Cr	1	Mo	1-10
Cu	10	Pb	1
Fe	1	Si	1-10
Mg	1	Ti	1-10

Elements sought and not detected:

Ag, As, B, Ba, Be, Bi, Ca, Cd, Co, Gs, Ge, Hf, Hg, In, Ir, K, Li, Mn, Na, Nb, Ni, Os, P, Pd, Pb, Rb, Re, Rh, Ru, Sb, Sn, Sr, Ta, Te, Ti, V, W, Zr, Zn.

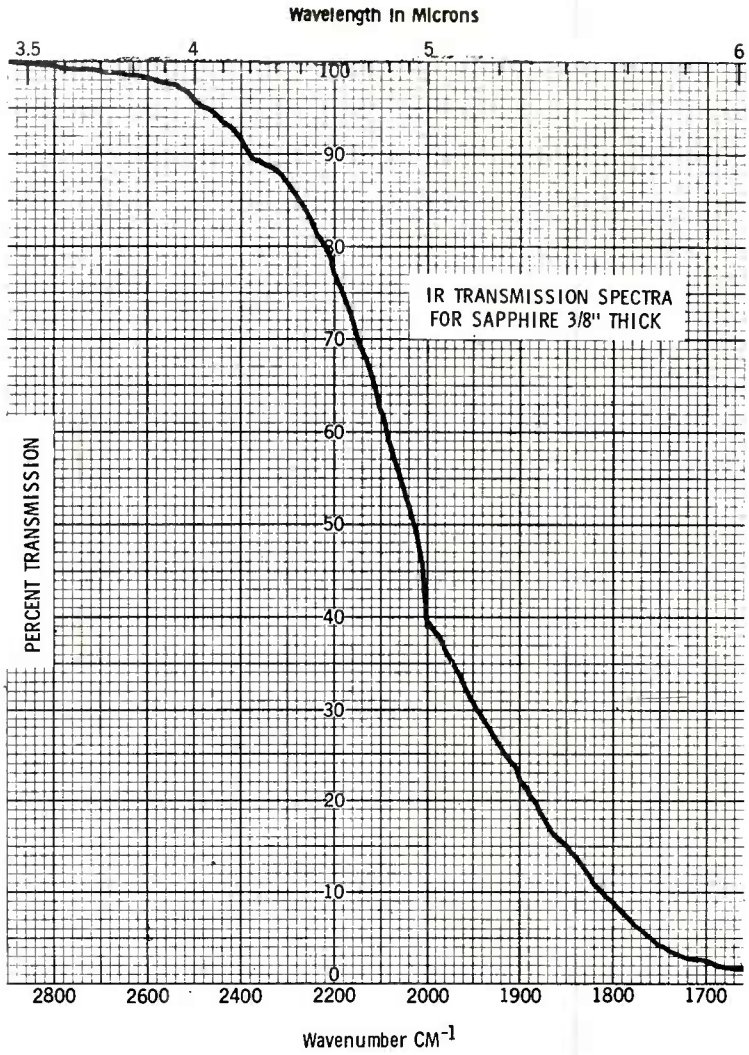


Figure 12. Infrared transmission spectra for sapphire 3/8" thick.

SUMMARY

A 20"-diameter vacuum furnace was received, installed, and utilized successfully to fabricate large single crystals of sapphire. The specifications for the furnace are included in the appendix.

A procedure was established to grow sapphire single crystal, up to 8 inches in diameter. A 12-inch-diameter single crystal sapphire was grown which cracked on cooling. This crystal exhibited the high clarity and high purity shown in Table 2. Progress in crystal growth capability is shown in Figure 13 by the increase in the size of the crystals. These sapphire crystals were also examined by observing X-ray pendelloesung fringes and comparing them with those from sapphire single crystalline material grown commercially. The quality of the gradient furnace-grown sapphire was the highest thus far observed. As to long-range crystal perfection, sapphire disks were examined polariscopically, revealing no subgrains. In fact, for the first time, 1/2"-thick sapphire disks over 6 inches in diameter have been cleaved, attesting to the degree of perfection of the sapphire crystals achieved during this investigation. Micrographs show cleaved surfaces with a step separation of over 1 millimeter. Sapphire single

Figure 13. Progress of sapphire single crystal growth.

crystals were grown without the "veiling" (Figure 11) which usually occurred in the finished crystal grown during the early phase of this project. Neutron activation analyses performed on these crystals revealed impurity levels of less than 10 parts per million. An infrared transmission curve was taken, utilizing this material, which showed a loss of less than 2% below 3.8-mm wavelength (Figure 12). This sapphire material showed great promise as a potential laser window material for applications at ∿3 μm due to its increased transmission of infrared with increasing quality of sapphire, and because of the superiority of its other physical properties as compared to other laser window material (Table 3). Sample sapphire windows were made for laser window applications and tested by TRW Corporation,[11] where they successfully survived initial tests as a window candidate for the HF and DF laser.

Table 3. MECHANISMS OF LASER WINDOW FAILURE

Material	Mechanical $\Delta P \longrightarrow$ Failure Flexural Strength $\times 10^3$	Thermal Shock		Thermal Lensing at 3 μm		Pressure Lensing Modulus $E \times 10^6$
		ΔT_c, °C	$\alpha \times 10^{-6}$	$dn/dt \times 10^{-5}$	$\beta \times 10^{-3}$	
Sapphire	>50	170	5-6.7	1	3	50
CaF_2	4	240	20-24	-0.6 - -0.12	3	18
SrF_2	Approx. 4			-1.2	3	15
BaF_2	Approx. 4		18-20	-1.7	3	--
Si	<50		4.2	3.9	1	19
Ge	<50		5.5-6.1	27.7	3	15
LiF	2-5.2		32-37	-1.6	3	9.3
CdTe	Approx. 5		4.5-6.2	10.7	2	5.3
MgF_2	7-15		13-19		5	20
GaAs			5.7	14.9	9	12

Note: Shaded areas are unacceptable values for laser application.

11. WONG, G. E., and WITTE, R. S. *Adsorption Measurements at DF Wavelength.* TRW Corporation, Report 28553.75.4351.2-157, November 1978.

An inexpensive surface finishing process for transparent armor which resulted in substantial savings in machining and polishing cost was developed by A. D. Little, Inc.[5] Sapphire made by this technique has been ballistically tested. The tests indicated no deleterious effects due to the sapphire being dipped into glass, i.e., the ballistic results were the same as those found for the uncoated sapphire.

APPLICATIONS

Due to the decreased processing costs and the availability of large-diameter sapphire single crystals with variable shapes, and because of its rather unique physical properties (Table 3), resistance to extreme moisture, scratch, and rain erosion, excellent thermal conductivity (K - 0.06 cal/cm sec °K),[12] etc., sapphire is emerging as an extremely attractive material for many Army applications. A partial list of current and potential applications follows:

Transparent Armor (up to 18" in diameter)

1-mm High-Power Laser Windows (no limit on diameter)

Electronic Substrates (Si on sapphire)

Electro-Optical Applications

Acoustic Surface Wave Device - substrate (AlN on sapphire)

Optical Windows - arc lamps

Reflex Sights

Scratch-Resistant Instrument Windows and Lens Caps

Gas Bearings for Inertial Guidance Systems.

It should also be noted that this advancement in the state-of-the-art in the growth of large single crystals of sapphire has resulted in furthering the technology of the growth of crystals of other materials by this process, e.g., yttrium aluminum garnet (YAG), silicon, etc. Techniques developed during the course of this project have been and will be beneficial in the processing of these crystals (cutting, polishing, coring, inspecting, etc.).

It should be further remarked that the scale-up phase of this project to proceed from growing 6"-diameter single crystals to 12" to 18" need not stop at this level. It is conceivable that scale-up can be extended further with the solution of technological problems, such as crucible fabrication, etc.

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12. SAHAGIAN, C. S., and PITHA, C. A. *Compendium on High Power Laser Window Materials.* AFCRL-72-0170, Bedford, MA, 1972.

APPENDIX. SPECIFICATIONS FOR VACUUM GRAPHITE RESISTANCE FURNACE

Requirements

High temperature (2500°C) graphite resistance furnace with a 20"-diameter, 20"-high vertical heating element. The furnace will include the following.

1. Furnace shall be fully water-jacketed 304 stainless steel chamber.
2. Jail Assembly - A 304 stainless steel chamber to house the insulation and heat zone.
3. Heat Zone
   a. Graphite felt insulation
   b. Heating element - slotted high purity graphite.
4. Power Supply - 3-phase saturable core reactor or silicon-controlled rectifier and step-down transformer.
5. Instrumentation
   a. Temperature control system: programmer, controller, recorder, and pyrometer
   b. Vacuum gage.
6. Vacuum System
   a. Mechanical vacuum pump
   b. Filter.

Detailed Construction

1.0 Furnace shell - Cylindrical with work port top and bottom.

1.1 Furnace shell position - Vertical with bottom work port at least two feet above floor level.

1.2 Furnace ports - The top will open to the full diameter of the chamber. The cover will be water-jacketed 304 stainless steel designed so that the top can be fully opened without optional equipment. A 1"-diameter sight port (on a 3/4" opening ball valve) will be positioned in the middle of the top closure.

The bottom work port will be 20" ID. The bottom cover will have a 4" ID entrance port in the middle with an O-ring groove and tapped bolt circle.

Three jacketed sight ports, 1" diameter, will be located on the side of the chamber so that the middle of the zone can be observed at 6", 10", and 14" heights. The sight glass will be on manual ball valves with a 3/4" opening.

A manifold with ports for a thermocouple gage, inert gas inlet, vent, ion gage, pressure gage, and pressure relief valve will enter near the mid-position of the chamber.

A jacketed port onto which a 12" diffusion pump can be attached will be located on the chamber. A water-cooled cover with a vacuum valve and line for the mechanical vacuum pump will cover this port.

1.3 Furnace interior - The interior surface will be smooth with no blind tapped holes or internal water connections.

2.0 Jail assembly - Cylindered to fit in furnace shell.

2.1 Ports - The top will open to the full diameter. The bottom will have a 3" hole in the center. Sighting holes will be located on the side to correspond to the sight ports in the furnace shell.

2.2 Alignment pins - The interior chamber will have alignment pins or holes on the bottom so it can always be placed in the same position in the furnace shell.

2.3 Handles - The chamber will have handles so it can be easily removed or placed in the furnace with a lift.

3.0 Heat zone - Cylindrical vertical 20" diameter × 20" high.

3.1 Heating element - Split graphite heating element 20" ID × 20" long, with uniform hot zone in the middle 12" capable of maintaining 2500°C, designed for easy replacement. The impedance will be matched to the power supply and be uniform on the entire element. The graphite power studs will be near the top of the element. The studs will be easy to attach to the element and will not introduce large stress.

3.2 Muffle tube - Cylindrical tube approximately 3/8" wall thickness with 3/4" clearance ance from element.

3.3 Insulation - Layers of graphite felt in excess of 3" on top, bottom, and sides.

3.4 Hearth plate - 1/2"-thick graphite disk 3/4" below heating element with 1-1/4"-diameter hole in the middle to fit the full diameter of the muffle tube. It will be supported by three graphite tubes extending through the insulation to a graphite plate on the bottom of the jail assembly.

3.5 Sight tubes - Cylindrical graphite tubes with 3/4" ID × 1" OD.

4.0 Power supply - 3-phase, 480-volt, saturable core reactor or silicon-controlled rectifier and step-down transformer.

4.1 Power requirement - The power supply will be sized so that at least 80% of the rated power will be utilized at 2500°C.

4.2 Safety gear

a. Appropriately sized shunt trip circuit breakers will be supplied for the furnace power supply.

b. Fully interlocked controls for power supply.

5.0 Instrumentation

a. Temperature control system: programmer, controller, recorder, and pyrometer

b. Vacuum gage

c. Safety gear.

5.1 Programmer - Shall be capable of adjusting the furnace temperature as a function of time. Process program will be displayed. The probe following accuracy shall be ±0.25% of span in static condition. The probe following repeatability shall be ±0.5% of span. Programmed cycle shall be adjustable to any time interval between 3 to 36 hours. Programmer shall include four programmable "on-off" function switches; a 48-hour timer shall also be provided to operate with the programmable "on-off" function switches.

Twenty blank programs shall be included.

Output of the programmer shall interface directly with the temperature controller defined in paragraph 5.2.

5.2 Temperature controller - Controller shall be supplied to provide for control of heating and cooling. It shall accept the signal from an optical pyrometer to control the temperature between 1800°C and 2100°C. The output will interface with saturable core reactor or the silicon-controlled rectifier. It shall be of solid state design with a sensitivity of 5 microvolts and a response time of less than 100 milliseconds.

The specification shall be:

a.	Input calibration	0 to 50 mV
b.	Resolution	1/1000
c.	Auto proportional band adjustable	0.1 to 5 mV
d.	Auto reset adjustable	0.1 to 10 repeats/minutes
e.	Auto rate adjustable	0 to 4 minutes
f.	Output limiter adjustable	10 to 100% of output
g.	Operation modes	manual, setpoint, programmable.

The controller shall have deviation and output meters to monitor deviation from setpoint and output of the controller.

The controller shall interface with the programmer described in paragraph 5.2.

5.3 Recorder - A 2-channel with 25 to 50 and 10 to 35 mV range. Eight-inch minimum width strip chart recorder shall have a response time of 3 seconds (full scale), automatic junction compensation chart speed of 3 inches per hour. Accuracy 0.3% span, repeatability 0.15% span.

5.4 Automatic radiation pyrometer:

a.	Special response	0.6 to 1.0 micron
b.	Focus range	18 inches to infinity
c.	Target diameter	less than 0.1 at 18 inches
d.	Calibration	1800°C to 2100°C
e.	Ambient temperature	40°F to 100°F
f.	Output (black body target)	50 mV full scale
g.	Repeatability	0.5% of full scale
h.	Accuracy	1% of full scale
i.	Linearity	better than 0.5% span

The radiation pyrometer will be supplied with mounting fixtures for permanent installation on the furnace.

5.5 Power sensor - Output, 0 to 50 mV; repeatability, 0.5% full scale; accuracy, 1% full scale; linearity, 0.5% full scale.

5.6 Vacuum gage thermocouple - Gage for monitoring vacuum in chamber and at the mechanical pump.

5.7 Meters and enclosure - Separate load voltage, current, and power meters shall be provided with the system.

The control system shall be mounted in an enclosure complete with side panels and doors.

5.8 Safety gear

a. Flow switches that will terminate furnace power when the water flow is insufficient.

b. Vacuum switch that will terminate furnace power when the pressure is greater than 1 torr.

c. Safety release valve will vent furnace when it reaches 15 psig.

6.0 Vacuum system

6.1 a. Mechanical pump with sufficient capacity to pump from atmospheric pressure to 0.1 torr in 10 minutes.

b. Ultimate vacuum at room temperature should be 50 microns.

c. Pressure rise due to leakage less than 40 microns per hour at 50 microns.

6.2 Filter to prevent contamination from entering the vacuum pump oil.

6.3 Vibration damper

a. The pump will be mounted on a vibration damper.

b. A flexible section vacuum line will be located between the vacuum pump and the vacuum valve to eliminate vibration to the furnace.

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The growth of large single crystal materials opens up many possibilities for new applications of ceramics, such as in the case of transparent armor, lasers, and laser windows. Single crystals of sapphire have been made by many techniques, the best known probably being the Verneuil method for the production of jewels and bearings. Until recently, the largest crystals of sapphire were probably those obtained by the Czochralski technique where crystals up to three inches in diameter were grown. At the Army Materials and Mechanics Research Center (AMMRC) a coordinated in-house effort has been underway to develop an improved approach to the growth of large uniform single crystals of sapphire. The main effort centered about a research and development project on the gradient furnace synthesis of advanced ceramics.

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